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Assessing Biodegradability of Hydraulic Fluids using Bio-kinetic Model

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ABSTRACT

Throughout the industrial age, natural resources have been turned into products no longer recognizable by microorganisms and enzymes converting natural substances into their basic building bocks. To make more environmentally friendly products, significant efforts are being made to develop biodegradable materials and technologies fully compatible with the environment. One of concerns in these efforts is the evaluation of their biodegradability in laboratory environments within a short period. Currently, several ASTM and OECD biodegradation test methods are available for determining this environmental property, but they take a long time (28 days) and special biological knowledge is required. To resolve this problem, a bio-kinetic model was developed to predict the biodegradability of hydraulic fluids. This report presents how to develop a bio- kinetic model and its results are discussed in a comparison with modified Sturm test (ASTM D 5864) and Aerobic Closed Respirometer test (ASTM D 6731).

INTRODUCTION

Petroleum based hydraulic fluids have been used in various hydraulic systems for many decades. These fluids are usually toxic and not readily biodegradable. A common factor in most hydraulic systems is the potential for leakage and the possibility of spillage of fluid during storage and use. The generation of the hazardous wastes by petroleum based or synthetic fluids results in both short and long term liabilities in terms of costs, environmental damage, and mission performance. Because of their poor environmental properties, there has been an increasing interest to use Environmentally Acceptable (EA) Hydraulic Fluids in environmentally sensitive areas such as construction, forestry, and agricultural. EA hydraulic fluids are currently defined as non-toxic and readily biodegradable products[1]. These fluids are originally formulated with renewable oils such as rapeseed, sunflower, corn, soybean, canola, and synthetic esters. These types of oils are currently considered less toxic and more biodegradable than conventional hydraulic fluids. One of concerns in these EA fluids is how to evaluate their environmental properties such as the biodegradability under laboratory environments.

Biodegradation is a natural process caused by the action of microorganisms, in the presence of oxygen, nitrogen, phosphorous, and trace minerals. Organic pollutants can support microbial growth and are converted into a series of oxidation products that generally conclude

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with carbon dioxide and water. In a previous study, it was found that the inherent biodegradability of a lubricant depends to a large extent upon its molecular structure and composition[2]. Typically, straight-chain aliphatic compounds (i.e., alkanes, cycloalkanes) are easily degraded. Simple aromatic compounds are usually slowly degradable and contain toxicity. Polymetric materials are among the most resist to microbial attack. It is also known that oils derived from renewable resources are more biodegradable than petroleum based oils. In addition, the water solubility or dispersability of petroleum based products, their molecular sizes, pH of solution, types of materials, temperature, total dissolved solids, and their toxicity, affect their biodegradability. Among them, it is strongly believed that the molecular structure and low toxicity may play a major role in the biodegradation of lubricant products.

A number of biodegradation tests have been developed to standardize evaluating the biodegradability of lubricants. These biodegradation tests were designed to determine the degree of aerobic aquatic biodegradation of fully formulated lubricants or their components on exposure to inoculums under controlled laboratory conditions. All of these tests can measure the biodegradability of lubricants based on oxygen uptake or carbon dioxide production of microorganisms (inoculums). Recently, ASTM D-2 Subcommittee 12 on Environmental Standard of Lubricants has developed several biodegradation test methods (ASTM D 5864[3], ASTM D 6731[4], ASTM D 6139[5]) for evaluating the biodegradability of petroleum based and renewable based lubricants. Their test procedures are very similar to each other and simulate the biodegradation process used in the waste treatment center. But these tests require a long testing time (28 days), the knowledge of microorganisms, and skilled manpower in some cases. In addition, they have very poor test precision due to the various and multiple inoculums sources. For these reasons, they are very difficult to use in petroleum laboratories for assessing the biodegradability of lubricants.

To resolve these difficulties, a new Bio-kinetic model has been empirically developed to predict the biodegradability of lubricants using a modified ASTM compositional analysis technique and the fundamental microbiological theory[6]. This bio-kinetic model requires compositional analysis data of lubricants and some of formulation information related to the types of base oils used in the lubricants. The advantages of this model is (1) its predictable capability for the biodegradability of lubricants within a day and (2), its excellent correlation with results obtained from ASTM D 5864 (Modified Stun Test). To further validate this model, a correlation study was conducted using various types of hydraulic fluids including renewable based fluids with two popular biodegradation tests (Modified Stun test, Respirometer) and CES Company's CO_2 evolution test[7]. This comparison study is essentially needed to determine whether a conventional biodegradation test or the bio-kinetic model can serve as a screening test for assessing biodegradability of fluids.

BIO-KINETIC MODEL

A bio-kinetic model is a new methodology to predict the biodegradability of a lubricant. This model has been developed based on the fundamental microbiology theory, and the compositional analysis of lubricant. This bio-kinetic model does not require any biodegradation test apparatus and inoculums. It can predict the biodegradability of lubricant within a day, and has a good correlation with the ASTM D 5864 biodegradation test using the round robin sample for ASTM D 5864[8]. The bio-kinetic model is described as follow:

$$B(t) = B(1) + \frac{0.49}{\ln(6.8 \times ECB^{-2.38})} \ln t$$

$$where \ :$$

$$ECB = \sum_{a}^{b} (\eta_a C_a + \eta_b C_b)$$

$$B(1) : 0.01$$

B (t) is expressed as a cumulative biodegradability of sample for elapsed time t. B (1) was found to be close to the first day of biodegradation which can be determined experimentally. The Effective Composition for Biodegradation (ECB) is sum of all saturate and ester fractions. C is represented by amount of composition of each type of oil and η is its ECB coefficient.

In this bio-kinetic model, the ECB value can be calculated using the ASTM D 2549, Separation of Representative Aromatics and Nonaromatics Fractions of High-Boiling Oils by Elution Chromatography. The schematic diagram of this test apparatus is shown in Figure 1. The test method tends to provide information on the separation and determination of representative aromatics and non-aromatics fractions from the petroleum based hydrocarbon oils using a chromatographic column. In a trial test, it was found that this procedure was not applicable to renewable based oils due to their inseparability problem. For this reason, some modification was made to analyze renewable products and its changes that are listed in Table 1. Mainly, the modified procedure has a four-step process instead of a three-step process. The first step eluted non-aromatic materials using a pentane solvent. Then, non-polar based aromatics were eluted using a mixture of 50 % pentane and 50 % toluene instead of diethyl ether. In step 3 process, the esters or related products were eluted using a diethyl ether. The polar-based aromatics were eluted in the step 4 process.

The modified procedure was designed based on the dielectric constant of solvents and their eluting characteristics. It was fully evaluated using known samples prior to the tests. The major advantage of this test is its small sample size requirement (2g), and its good test precision. Also, it was found that this procedure can not differentiate between some chemicals that are eluted by same solvent such as mineral oil and polyalphaolefin (PAO) oils, or esters. In this case, the identification of oils can be directly obtained from oil manufacturers or through additional tests (i.e., Gas Chromatography (GC) or Infrared Spectroscopy (IR)).

Generally, the biodegradability of hydrocarbon based oils depends on the types of materials and chemical structures. For this reason, an adjustment was essentially needed for this compositional analysis to determine the effective composition for the biodegradation (ECB) of lubricants. Table 2 lists the ECB coefficient for each type of oil. These coefficients were calculated based on the data obtained from biodegradation tests and range from 1 for the renewable ester to 0.01 for the Petroleum based ester type. In this calculation, ECB is sum of all fractions of saturate and esters except for all aromatic fractions that are considered as toxic materials.

TEST SAMPLES

The hydraulic fluids are currently formulated with various types of oils and additives to meet specific applications. The four most common types of hydraulic fluids are mineral oil, synthetic esters, polyalphaolefin (PAO), and vegetable oils. Currently, these types of fluids are widely used in conventional hydraulic systems. For the study, seven typical hydraulic fluids were selected including canola cooking oil as a positive control. They consists of four vegetable-based fluids (i.e., two canola, soybean, rapeseed), a PAO- based fluid, a mineral based oil, a polyol-ester based fluid, and a PAO-ester blended fluid. These samples were fully formulated by the lubricant companies with different types of base oils, viscosity grades, and chemical additives. All of these fluids are currently being used for the hydraulic applications and listed in Table 3.

TEST RESULTS AND DISCUSSIONS

To assess the bio-kinetic model, eight different types of hydraulic fluids were tested according to three conventional biodegradation tests (i.e., ASTM D 5864, ASTM D 6731, CES CO_2 evolution Test). Although these tests use identical test solutions and inoculums, their measuring techniques of the biodegradability are not same. All of these biodegradation tests require a 28 days exposure period. Figure 2 shows the typical biodegradation profiles of the conventional ~~biodegradation~~ tests and bio-kinetic model for canola based EA fluid (Code C). Table 4 summarizes the biodegradation test results of the tested fluids. Data obtained from bio-kinetic model are also presented in this table to provide a comparison with the conventional biodegradation tests. It is noted that these results were determined based on triplicate test results in order to increase reliability of test data and are expressed as percentage of the maximum (theoretical) carbon dioxide evolution of each sample or oxygen consumption of microorganisms in each test. In these tests, the biodegradability of hydraulic fluids ranges from 30 to 90 % for 28 days. It is quite evident that the EA hydraulic fluids provided a higher biodegradability than a conventional petroleum based hydraulic fluid. In addition, a 2 cSt PAO based fluid also provided a good biodegradability similar to those of vegetable - based fluids. Data obtained from the bio-kinetic model provided ~~a~~ good correlation with those of the conventional biodegradation tests.

The Bio-kinetic model is able to calculate the biodegradability of fluids based on the compositional analysis within a short period. This model was originally developed based on the carbon conversion of the test sample in a similar approach to that of ASTM D 5864 and its ECB value. The ECB of each fluid was actually measured using the modified ASTM D 2549 compositional analysis technique that provides an excellent test precision. This technique eluted four different types of chemical compositions from a fluid. It consists of non-aromatics compounds such as saturates (mineral oils, PAO), non-polar based aromatics, ester groups, and polar based aromatics. Generally, the aromatic compounds tend to reduce the biodegradability of fluids due to their structure and toxicity. In this bio-kinetic model, the aromatic compositions were ignored for ECB calculation. Table 5 shows the compositional analysis of tested fluids and their ECBs. It clearly indicated that each fluid has a different chemical composition and ECB. The high composition of ester gave a high ECB value that is reflected to the high biodegradability of that fluid. In the other case, it was found that the 2 cSt PAO fluid (Code E) composed of 98.8 % saturates and its ECB coefficient is 0.8 that is much higher than that of mineral oil (0.3). Table 5 shows that its biodegradability ranges from 61.3 to 78.4 % in four biodegradation tests. It appears that this analytical technique is not only providing good biodegradability differentiation, but how one can improve the biodegradability of fluids based on their compositional analysis. also information regarding

ASTM D 5864 biodegradation test is a version of the Organization for Economic Co-Operation and Development (OECD) 301 B, Modified Stun Test that closely simulates the waste water biodegradation conditions. This test was designed to determine the degree of aerobic aquatic biodegradation of lubricants on exposure to inoculums under laboratory conditions. In this test, the biodegradability of a lubricant is expressed as the percentage of maximum carbon conversion under well-controlled conditions for a period of 28 days. The test apparatus consists of four separate units: the free carbon dioxide air system, biodegradation batch reactor, a carbon-dioxide collector and a titrator ~~and~~. The test apparatus can be easily constructed by the ~~any~~ testing laboratory. Most of these components involve regular laboratory glasswares (i.e., flasks, etc.). This test apparatus is considerably less expensive than the other types of biodegradation tests (i.e., Respirometer). For the biodegradation test, the five test stock solutions for the test medium were prepared to provide nutrition for microorganisms. These stock solutions were Ammonium Sulfate solution, Calcium Chloride solution, Ferric Chloride solution, and Magnesium. It should be noted that these solutions do not contain any carbon material in order to avoid an extra source of carbon dioxide production. Canola cooking oil was used as a positive controller to verify the microorganism's performance during the test. In addition, the initial carbon content of the test lubricants was measured to establish the maximum theoretical biodegradability of that lubricant. The sewage microorganism from a local waste water treatment plant was used as inoculums in this test. The test was performed for 28 days under dark environment, and the titration for carbon-dioxide cumulation was performed every day for the first ten days and then every other day for the remaining 18 days or until a plateau of carbon-dioxide evaluation was reached. This test has been widely used for many decades by industry and is considered as a reliable biodegradation test. The disadvantages of this test are its poor precision due to the various of inoculums sources and unknown microorganisms' behavior as well as long test times. In addition, this test requires skilled manpower and sufficient laboratory space. Currently, the ASTM D 6046, Hydraulic Fluids for Environmental Impact specifies that the persistence designation of the readily biodegradability is Pw 1 and must be greater than or equal to 60 % of CO_2 evolution in 28 days. Compared with ASTM D 5864 test, the bio-kinetic model does not need the biodegradation test apparatus and inoculums. Table 5 shows their data to be very equivalent to each other. In addition and more importantly, the bio-kinetic technique can reduce the test time from 28 days to 1 day.

ATSM D 6731 Biodegradation test is another version of OECD 301F, The Manometric Respirometry Test and is known as the modified Biochemical Oxygen Demand (BOD) Test. This closed respirometer test was also designed to determine the degree of biodegradability of lubricants or their components in an aerobic aqueous medium on exposure to an inoculums under laboratory conditions. Unlike the ASTM D 5864 test, the biodegradation of a lubricant is determined by measuring the oxygen consumption of microorganisms instead of the carbon conversion of the test sample. This approach was developed based on ~~one of~~ the assumption~~s~~ that a large amount oxygen uptake of microorganisms indicates more microorganisms' growth or generation and takes more carbon conversion of the test sample leading to carbon dioxide production by an enzyme process. For this reason, the respirometer test is currently considered as an indirect biodegradation test of lubricants, and its biodegradability is expressed as the percentage of maximum oxygen consumption under well-controlled conditions for a period of 28 days. The test apparatus of this test method is a well designed automatic system and requires less manpower. In this test, the oxygen consumption is measured based on the pressure drop of the manometer, which produces a signal that results in the electrolytic generation of oxygen. The sample and medium preparation is almost identical to ASTM D 5864 test sample. The advantages of this method are less manpower required and its closed system that is suitable for evaluating the biodegradation of volatile lubricants. The disadvantage is its indirectly measurement of biodegradation of lubricants and its poor test precision. Because of its measuring technique and the cost of test apparatus, this method is not widely utilized within industry. Currently, the ASTM D 6046, Hydraulic Fluids for Environmental Impact specifies that the readily biodegradability of fluid (classification: Pw1) in this test must be greater than or equal to 67 % of O_2 consumption in 28 days. This value is a little higher than that of ASTM D 5864 test because of its total oxygen consumption of microorganisms including biomass production. In this study, the test results were also very comparable with those obtained from the bio-kinetic model. The CES respirometer apparatus has an option to measure carbon dioxide evolution directly during a respirometer test. In this option, the microorganisms consume oxygen from the respirometer and produce carbon dioxide. Then, this carbon dioxide is trapped in an absorbent solution (i.e., Sodium Hydroxide) and measured by a CO_2 sensor instead of a titration. This technique is very similar to that of ASTM D 5864 test, while the test results are much closer to those of ASTM D 6731 because of its closed system that can minimize the ~~lost~~ loss of carbon dioxide during the biodegradation process. This optional test is not yet developed as a standard test method, but can be considered as a closed automatic ASTM D 5864 test. The advantage of this test over the ASTM D 5864 test is in its automatic system, but it still has a test precision problem. Generally, the closed biodegradation system produces a higher biodegradability of the tested fluids than those of semi-open system.

To determine the correlation between biodegradation tests including bio-kinetic model, the data ~~were~~ analyzed to find the correlation coefficient (r^2) using a statistic method. Table 6 shows ~~that~~ their correlation coefficient were found to ~~be~~ ranged from 0.8 to 0.95. It appears that the bio-kinetic model gave a good agreement with the selected biodegradation tests. Typically, the bio-kinetic model gave an excellent correlation to both ASTM D 6731 and ASTM D 5864 tests. The CES biodegradation test also gave a good agreement with those of ASTM D 5864 test. Table 5 clearly shows that data obtained from the biodegradation tests correlated each other. A typical correlation curve is shown in Figure 3. Therefore, the bio-kinetic model can be also used to predict the biodegradability of hydraulic fluids within a short period. Currently, ASTM D-2 Subcommittee 12 on Environment is reviewing this bio-kinetic model to adopt as an ASTM biodegradation test method.

CONCLUSIONS

On the basis of the work completed to date, the bio-kinetic model was fully evaluated using eight different types of hydraulic fluids and compared with the conventional biodegradation tests. This bio-kinetic model provided excellent correlation with results obtained from the conventional biodegradation tests. In addition, it reduced significantly testing time from 28 days to one day and can easily predict the biodegradability of fluids without the handling of microorganisms. Therefore, the bio-kinetic model can be used for predicting the biodegradability of fluids as a screening test. The results of this study are summarized with the following findings:

1. The Bio-kinetic model gave good correlation with the conventional biodegradation tests (i.e., ASTM D 5864, ASTM D 6731, CES CO_2 evolution test). Their correlation coefficients (r^2) were found to be from 0.8 to 0.95.
2. ASTM D 5864 biodegradation test gave a good agreement with ASTM D 6731 respirometer test, but its results were slightly lower than those of the respirometer due to its manual operation and semi-opening system. In addition, it provided ~~a~~ very poor precision like the other biodegradation tests due to the various inoculums ~~sources~~.
3. CES biodegradation test is an optional CO_2 evolution test of CES closed respirometer and gave a good agreement with ASTM D 5864 test, but its results were very similar to those of the respirometer test. It appeared that the ASTM D 5864 test tends to lose some of CO_2 production due to its semi-opening test apparatus. Thus, the CES CO_2 evolution test can be considered as an automatic ASTM D 5864 biodegradation test.
4. Modified ASTM D 2548 test was able to analyze the compositions of fluids and provided an excellent test precision. But, it can not differentiate between mineral oils and PAO oils and between renewable ester and non-renewable ester. It requires pre-information of base oil types from oil companies or other techniques (i.e., IR, GC).
5. The effective composition for biodegradation (ECB) gave a good correlation with the biodegradability of fluids. In addition, it can provide vital information on how to improve the biodegradability of fluids.

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Table 1. Modified ASTM D 2549 Procedure

Process	Modified ASTM D 2549	
Step 1	Pentane	Non-aromatics (saturate, mineral oil, PAO)
Step 2	50 % pentane+50% toluene	Non-polar based aromatics
Step 3	Diethyl ether	Esters, acid, waxes
Step 4	Chloroform/Ethyl alcohol	Polar based aromatics

Table 2. ECB Coefficients for Oils

Lubricant	ECB Coefficient (η)
Mineral oil	0.3
PAO 2*	0.8
PAO 4*	0.6
PAO 6 or above*	0.4
Natural esters	1
Renewable based diester	0.8
Petroleum based ester types	0.01

* Viscosity grades for polyalpaolefin (PAO)

Table 3. TEST SAMPLES

Code	Type of base oil	ISO classification	Identification
A	Soybean	46	Commercial HF*
B	PAO 4 + diester	22	MIL-PRF-48170[8]
C	Canola	46	MIL-PRF-32073[9] Grade 4
D	Canola	46	MIL-PRF-32073 Grade 4
E	PAO 2	2	Commercial HF
F	Mineral Oil	100	SAE 15W-40
G	Rapeseed	32	Commercial HF
H	Polyol ester	22	MIL-PRF-32073 Grade 2
I	Canola	-	Cooking Oil

*Hydraulic fluid

Table 4. Biodegradation Test Results

Code	ASTM D 5864	ASTM 6731	CES CO_2 Evolution Test	Bio-kinetic Model
A	61	63.4	73.7	64.0
B	51.9	51.3	47.8	51.0
C	72.3	75.3	74.1	77.8
D	71.7	75.2	69.7	69.3
E	71.2	78.4	61.3	67.0
F	30.1	36.6	24.1	32.8
G	74.0	84.8	80.2	79.0
H	66	76.6	66.2	70.0
I	73.3	88.0	94.7	77.2

Table 5. Composition Analysis of Tested Fluids

Code	Alkanes or Saturate	Non-polar aromatics	Ester, Acid, or Wax	Polar aromatics	ECB*
A	24.5	2.2	67.9	5.4	0.75
B	68.7	2.9	27.7	0.7	0.55
C	3.49	2.57	90.56	3.38	91.6
D	17.53	1.32	77.45	3.69	0.82
E	98.8	0.47	0.05	0.7	0.79
F	82.5	13.5	1.4	1.7	0.26
G	0.07	1.00	92.95	5.98	0.93
H	17.5	1.32	77.5	3.7	0.83
I	0.18	1.6	90.7	7.5	0.91

* Effective composition for biodegradation

Table 6. Correlation Coefficients (r^2) between Biodegradation Tests

	ASTM D 5864	ASTM D 6731	CES CO_2 Evolution Test	Bio-kinetic Model
ASTM D 5864	1	0.92	0.8	0.95
ASTM D 6731	0.92	1	0.81	0.92
CES CO_2 evolution test	0.8	0.81	1	0.87

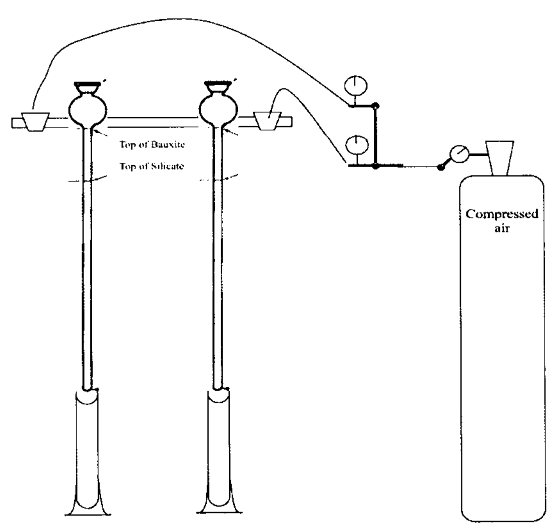

Figure 1. ASTM D 2549 Test Apparatus

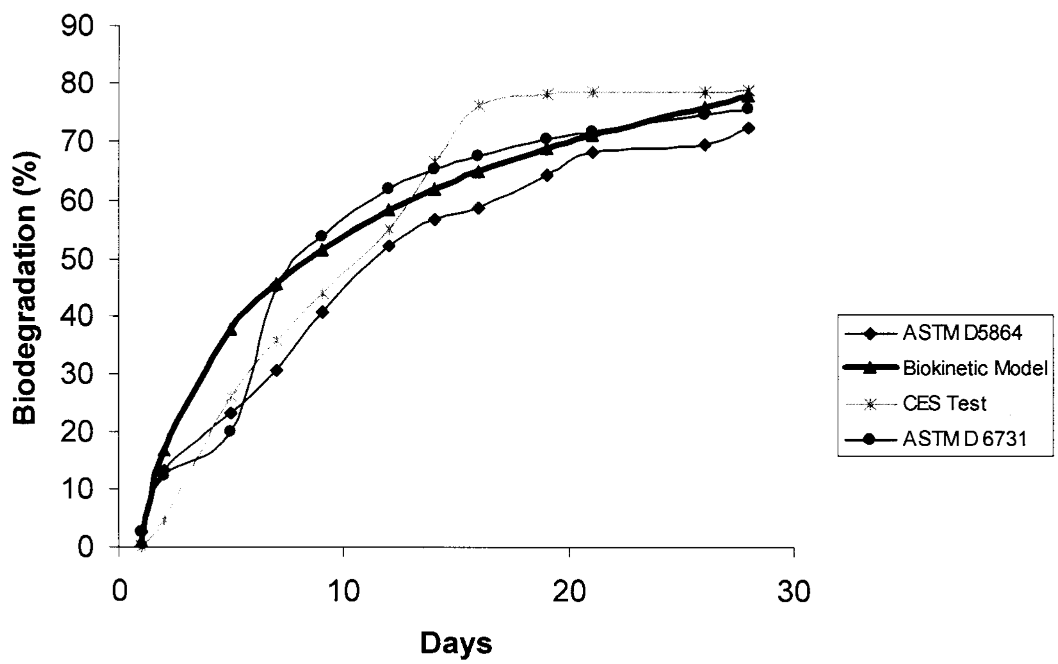

Figure 2. Biodegradation Profile for Canola based BHF

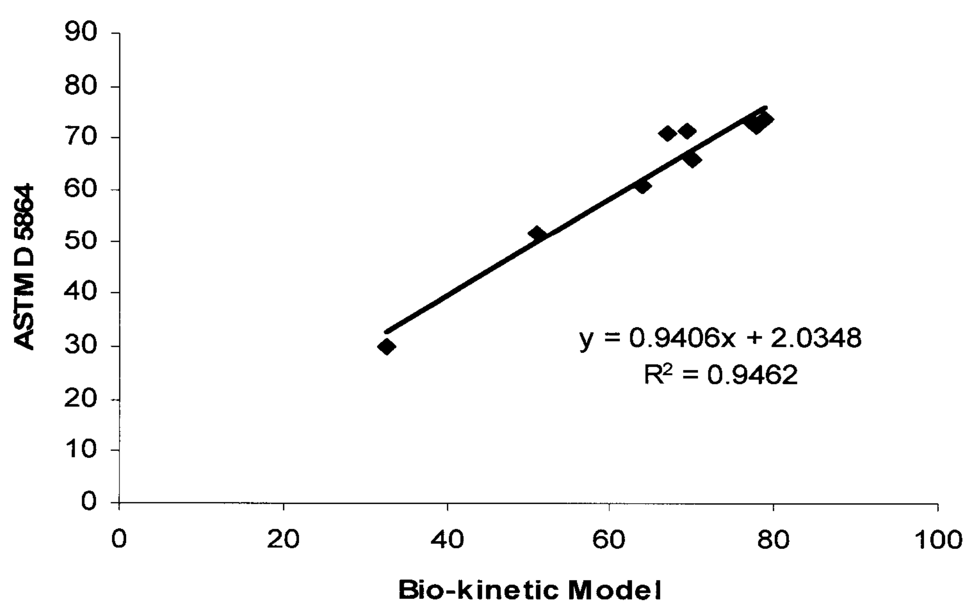

Figure 3. Correlation between Bio-kinetic Model and ASTM D 5864